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16. ABSTRACT

In routine surveillance operations, the radionuclide measurement of air discharged from an operating nuclear facility involves the entrainment of radionuclides on selective filter or absorptive media, and the determination of their gross beta activity. However, a more sensitive technique is required when specific analysis is desired for valid assessment of environmental radiation exposure, particularly when the gross beta activity levels are below 100 fCi/m^3. For measuring environmental levels of ^{106}Ru in air samples on glass fiber filters, a radiochemical procedure has been developed, tested and evaluated.

The method which involves digestion, distillation, and precipitation to separate and purify the radioruthenium has been employed in the analysis of air samples over the past year and a half. The method has been subsequently modified so that ruthenium, strontium and cesium can be measured sequentially on a single air filter.

Minimum detection limits (3σ counting error) have averaged <0.02 fCi/m^3 for ^{106}Ru and <0.01 fCi/m^3 for ^{137}Cs and $^{89/90}Sr$; chemical yields have been 75, 80 and 75%, respectively.

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SEQUENTIAL RADIOCHEMICAL ANALYSIS FOR RUTHENIUM, STRONTIUM AND CESIUM IN ENVIRONMENTAL AIR

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Abstract—In routine surveillance operations, the radionuclide measurement of air discharged from an operating nuclear facility involves the entrainment of radionuclides on selective filter or absorptive media, and the determination of their gross beta activity. However, a more sensitive technique is required when specific analysis is desired for valid assessment of environmental radiation exposure, particularly when the gross beta activity levels are below 100 fCi/m^3. For measuring environmental levels of ^{106}Ru in air samples on glass fiber filters, a radiochemical procedure has been developed, tested and evaluated.

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INTRODUCTION

CONTINUOUS surveillance of radioactivity in air provides one of the earliest indications of change in environmental fission product concentrations. This surveillance, which has been confined primarily to gross beta measurements and/or qualitative gamma emitter identification is primarily of interest in reporting unexpected changes in activity levels or nuclide composition. In order to assess total human radiation exposure from such activity, identification and quantitative measurement of specific radionuclides are required.

At the present time airborne radioactivity surveillance is performed at nuclear power stations by routine sampling of discharged air. The activity is entrained on selective or absorptive media over a specified period of time, so that it can be related to the stack discharge from the installation. When full-scale operation of nuclear fuel reprocessing plants becomes a reality, an increase in the airborne discharge of radioactivity from their stacks will result. Combined with this will be a new spectrum of radionuclides to be considered in the monitoring protocol for their collection, identification, and quantification in a variety of media. It would no longer be possible to relate activity increases to fission product inventory by means of gross measurements. This is particularly the problem since it has been ascertained that in addition to the expected fission products, higher concentrations of ^{129}I, ^{239}Pu and ^{106}Ru will be introduced into the air, soil, and vegetation (MAGNO *et al.*, 1970).

EXPERIMENTAL ANALYSIS

Airborne surveillance at the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, involves the collection of a sample on an 8 × 10 in. glass fiber filter over a 7–10 day period. The initial flow rate is 50–55 ft^3/min, but as particulate debris accumulates on the filter, the flow decreases to as little as 30–40 ft^3/min at the end. This collection period is many times longer than in normal surveillance network station operations and assures the accumulation of activity levels which are definitely above the minimum detection level (MDL). However, the activity levels in these filters are subject to unacceptably large errors.

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Table 1. Gamma activities in selected air filter samples, fCi/m³

Nuclide	MDL 3σ Counting Error	Spring 1974	Summer 1974	Autumn 1974	Winter 1974	Spring 1975	Summer 1975
^{144}Ce	0.8	69.8 ± 3.0	15.4 ± 1.0	5.6 ± 1.2	8.7 ± 0.8	35.1 ± 2.7	9.5 ± 1.0
^{141}Ce	0.3	2.3 ± 0.2	5.4 ± 0.5	0.5 ± 0.4	1.6 ± 0.2	1.1 ± 0.4	<0.5
^{131}I	0.2	<0.2	0.5 ± 0.2	<0.2	<0.2	<0.2	<0.3
^{125}Sb	0.4	3.5 ± 1.0	0.9 ± 0.5	0.3 ± 0.2	0.5 ± 0.3	2.4 ± 1.0	1.0 ± 0.5
^{7}Be	1.2	149.9 ± 5.0	100.4 ± 5.0	68.2 ± 1.5	63.3 ± 3.5	139.9 ± 5.0	73.7 ± 2.4
^{103}Ru	0.3	6.5 ± 1.0	3.3 ± 0.5	0.7 ± 0.2	2.8 ± 0.6	2.9 ± 1.0	0.5 ± 0.3
^{106}Ru	1.2	31.1 ± 2.0	6.2 ± 2.5	2.7 ± 1.5	3.4 ± 1.8	15.4 ± 3.9	3.6 ± 1.3
^{137}Cs	0.2	5.7 ± 0.5	1.6 ± 0.5	0.7 ± 0.2	0.8 ± 0.3	3.1 ± 0.5	1.5 ± 0.3
^{95}Zr	0.4	30.4 ± 2.0	7.1 ± 1.0	1.8 ± 0.5	7.6 ± 0.6	40.0 ± 1.7	3.2 ± 0.5
^{95}Nb	0.3	67.3 ± 3.5	9.6 ± 1.0	3.2 ± 0.5	15.6 ± 0.8	28.1 ± 1.0	4.1 ± 0.5
^{54}Mn	0.4	<0.3	0.4 ± 0.2	<0.3	<0.3	<0.3	<0.4
$^{140}Ba/^{140}La$	0.6	<0.5	2.4 ± 0.6	<0.5	<0.5	<0.5	<0.5

These 8 × 10 in. filters are folded to a 3 × 2 in. geometry then inserted into a plastic envelope, and gamma-scanned with a Ge–Li detector calibrated for this geometry. The predominant gamma photopeaks can be identified very readily, yet quantifying their activities is quite inaccurate. Similiarly, at this geometry, identification of some of the less abundant nuclides is uncertain and quantification impossible. In addition, the significant counting error precludes any assurance that a radiation exposure problem does, in fact, exist, when any calculation of dose is made from such data.

Gamma-spectral analysis results of representative air filters are shown in Table 1. Although other gamma emitters may be present, their identification must be tentative without further corroboration. The activity of these gamma emitters would not be much greater than instrument background and more apt to be close to the 3σ MDL counting error. In network surveillance operations where even less sophisticated instrumentation is in operation, the unidentified list would be much greater. These filters, collected during different seasons of the year were counted for 1000 min on the Ge–Li detector system and represent an average 7–8 day accumulation of activity removed from 20,000 m³ of air. The results indicate that, at certain seasons of the year, some of the nuclides are not much above the minimum detectable level of the instrument and that even with a long counting time, the 2σ counting error is a considerable fraction of the nuclide activity. Thus, for more realistic values which can be utilized in human dose appraisal, specific radiochemical analyses would be recommended.

METHODOLOGY

The procedures utilized to day for the analysis of ruthenium in water can be made applicable, with slight modification, to soil, biota and vegetation samples. The methodology normally includes steps that destroy interfering agents and oxidize the ruthenium to its volatile tetroxide state. Under controlled conditions, the ruthenium can then be distilled into caustic and purified as the metal (Harley, 1972; Kleinberg, 1954).

With glass fiber filters, however, much additional treatment was required before the analyses could be performed satisfactorily. After the high-vol. sampler has been in operation for a week or more, the glass fiber filter takes on a very darkened appearance which interferes with the initial oxidation of the ruthenium to the tetroxide. The procedure that was effective for analyzing 10–100 ml aqueous samples was ineffective on glass fiber filters, and further method evaluation was undertaken.

In the tests that followed, the stable ruthenium carrier was added uniformly to the filter which was air-dried overnight. To assure complete digestion with the sulfuric acid, the filter was cut into small strips before transferring to the digestion flask. During digestion, constant boiling of the 9 M H_2SO_4 was maintained throughout, and solid $KMnO_4$ was added in the oxidation step to assure complete conversion of the ruthenium to the tetroxide. For the distillation step, the large sample volume necessitated excess caustic in the trap to prevent loss of ruthenium due to a decrease in pH. The ruthenium procedure, as finally modified, was tested on air samples and without added ^{106}Ru tracer. The results

indicated good agreement between chemical and radiochemical yields—with an average of 75–80% for each.

Inasmuch as strontium and cesium analyses were also desired on the same filter, additional testing and modification of existing procedures was undertaken. Although the filter could have been divided into aliquots for each analysis, the near background activity levels of some of the nuclides dictated that the larger the sample size, the more valid the result.

The finalized sequential procedure is detailed in the Appendix. The initial steps define the ruthenium digestion and distillation; then, the aqueous fraction of the permanganate residue is treated to separate the cesium and purify it as the chloroplatinate; and finally the strontium in the MnO_2 solids is separated and subsequently purified as the carbonate. All three stable carriers are air-dried on the filter before analysis and the chemical yields that result, reflect the effectiveness of the lengthy procedure. Average recoveries for the sequential procedures have been 75% for the ruthenium, 80% for the cesium, and 75% for the strontium.

Table 2 compares the 3σ minimum detection limits obtained by overnight Ge–Li counting of the filter with those that would be obtained by a 400-min beta count of each nuclide radiochemically separated and filtered on a nylon ring and disc. It is obvious that when fuel reprocessing plants will be discharging ^{106}Ru in their stack effluents, the ability to detect such low levels with assurance becomes important.

The results of sequential analysis of air filters since January 1974 are shown in Table 3. These monthly samples were collected for about 7 days, and the flow through for each sampling period averaged 19,000 m³. Spectral and radiochemical analysis values for ruthenium and cesium are compared, and they are in good agreement. However, the 2σ gamma counting errors of 2×3 in. filters at these levels is >10%. At levels close to the MDL, these errors can exceed 50% even with overnight counting. Beta counting the separated planchet for 400 min involves <10% error and an MDL that is lower by a factor of 10. The ^{90}Sr values which were determined radiochemically over this same period ranged from 0.3 to 3.2 fCi/m³. For all nuclides, in addition to seasonal variations, the 1975 values are decreasing from their corresponding 1974 levels. This of itself would necessitate more sensitive monitoring techniques, and it has been shown that sequential radiochemical analysis will accomplish this end.

*Table 2. Comparison of minimum detection levels (3σ),fCi/m³**

Nuclide	Non-Destructive Spectral Analysis (Filter folded four times)	Radiochemical Separation (on nylon ring & disc)
^{103}Ru	0.3	0.05
^{106}Ru	1.2	<0.02
^{137}Cs	0.2	<0.01
^{90}Sr	---	<0.01

* These values have been determined by calculating the three-sigma (3σ) levels for the instrument backgrounds (Ge-Li detector and LBBC, respectively) when the samples were counted for 1000 minutes on the Ge-Li, and 400 minutes on the low-background beta counter.

DISCUSSION AND CONCLUSIONS

One of the goals of surveillance operations is to assess the effect of plant effluents on the environment and to determine their effect on the population. In the case of a nuclear fuel reprocessing plant wherein one of the nuclides of concern would be ^{106}Ru, air-monitoring techniques would be employed to discern whether unusual changes in activity levels occur in the course of daily operation. Because these changes may come about ever so slightly and nuclide species may be different, a gross activity of discharged air is not satisfactory.

A more sensitive analytical technique is required which would enable more accurate prognostications to be made regarding the existence of a radiation exposure problem. In addition, any uncertainty with respect to a large counting error would have to be minimized. The minimum detectable level for such a measurement must be extremely low in order to give credence to the results of the calculation.

A radiochemical method for ^{106}Ru in air has been improved so that ^{106}Ru, ^{137}Cs and ^{90}Sr can be analyzed sequentially on the same filter. With low levels of these activities observed in pre-operational sampling, it is imperative that as large a sample as possible be made available for analysis. Taking aliquots of a filter for separate analysis, increases the error of the result and introduces an additional sampling error in the sectioning of the air filter.

Table 3. Comparison of gamma spectral and radiochemical analysis** of air filters*

Sampling Period 1974	1000 m^3 Flow Through	fCi/m^3 ^{103}Ru Gamma Spectral Analysis	^{103}Ru Radiochem. Analysis	^{106}Ru Gamma Spectral Analysis	^{106}Ru Radiochem. Analysis	^{137}Cs Gamma Spectral Analysis	^{137}Cs Radiochem. Analysis	^{90}Sr Radiochem. Analysis
1/11-18	17.6	2.9	2.6	4.8	1.7	0.78	0.78	---***
2/1-8	17.0	2.4	2.6	3.5	4.0	0.81	0.81	---***
3/4-12	22.4	6.4	6.4	15.6	13.9	2.9	2.9	0.4
4/19-26	20.4	6.5	8.5	31.1	30.4	5.7	6.4	2.1
5/15-21	17.2	4.7	5.8	30.5	33.1	5.4	6.1	1.6
6/7-14	21.9	1.9	1.6	19.3	18.5	3.7	4.3	0.9
7/9-17	18.6	2.5	2.2	19.0	21.2	4.8	5.4	2.5
8/8-16	19.7	3.3	3.8	6.2	7.4	1.6	2.0	1.8
9/13-18	14.5	0.64	0.51	2.4	2.6	0.73	1.0	3.2
10/16-25	21.6	0.68	0.69	2.7	2.4	0.70	0.93	1.0
11/13-20	19.1	1.9	1.3	5.2	2.6	0.86	0.94	1.5
12/18-26	24.0	2.8	2.0	3.4	2.0	0.74	1.1	0.4
1975								
1/15-20	18.5	2.1	1.4	3.1	2.4	1.11	0.75	0.3
2/7-14	19.8	2.6	1.9	4.6	4.6	1.18	1.5	0.8
3/5-12	18.9	3.4	2.3	10.6	6.7	2.9	2.9	1.4
4/4-11	20.1	2.0	2.3	15.4	9.4	3.1	3.7	1.3
5/7-14	21.8	0.9	0.9	9.7	6.9	2.4	2.6	1.4
6/10-17	20.8	0.5	0.5	3.6	3.8	1.3	1.3	1.3

* The 2σ counting error for gamma counting the air filters ranged from 10-30%.
** The 2σ counting error for beta counting the separated planchet was 10% or less.
***Prior to March 1974, there were no radiostrontium separations on these air filters.

The sequential procedure, as delineated in this report, has been employed in the analysis of monthly air samples for the past 1.5 yr. The analytical method, which assures at least a 10-fold increase in sensitivity can detect 0.02 fCi $^{103}Ru/^{106}Ru$, 0.01 fCi ^{137}Cs and ^{90}Sr per m^3 of air. The sequential procedure assures chemical yields of 75, 80 and 75%, respectively.

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APPENDIX—SEQUENTIAL ANALYSIS OF RUTHENIUM, CESIUM AND STRONTIUM IN AIR FILTERS

Ruthenium analysis

(1) Remove the 8 × 10 in. glass-fiber filter from the sampler. Fold into a 2 × 3 in. rectangle and insert in a plastic bag. Label.

(2) After 5 days, to allow for radon decay, count the folded filter with a Ge(Li) detector to ascertain predominant photopeaks and to calculate gamma activities.

(3) After the spectral analysis, open filter and saturate with 2.0 ml ruthenium carrier by dropwise addition of carrier over entire filter. At the same time, add 1.0 ml each of strontium and cesium carriers. Allow to air dry thoroughly.

(4) Cut filter into small strips and transfer to a 1000-ml distillation flask, and add 500 ml 1:4 H_2SO_4.

(5) Digest for 1 hr at a gentle boil, stirring contents intermittently. Cool in ice bath.

(6) Connect the flask and the special ruthenium still to an air inlet and insert the delivery tube into a centrifuge bottle containing 50 ml 6 N NaOH in an ice bath.

(7) To distillation flask, add 25 ml 0.5 M $KMnO_4$, and immediately close system.

(8) Pass air gently through system to drive volatile ruthenium into the NaOH solution.

(9) Gently heat to boiling and continue heating for 10–15 min, or until distillation of ruthenium is complete. Turn off air, disconnect apparatus and reserve contents of flask for cesium and strontium analyses (Step 17).

(10) Add 5–10 ml ethanol to distillate, stir, boil to precipitate RuO_2 and cool. Centrifuge and discard supernatant.

(11) Suspend precipitate in 10 ml water containing 1 ml 6 N NaOH. Boil, cool, centrifuge and discard supernatant.

(12) Dissolve precipitate in 2 ml 6 N HCl, heating gently in a water bath to aid dissolution.
(13) Add 10 ml water and slowly add 100–200 mg magnesium metal in small portions, adding each portion when the previous addition has been oxidized.
(14) Boil until ruthenium metal has completely precipitated. Wash walls of tube and dissolve residual magnesium by dropwise addition of 12 N HCl. Cool, centrifuge and discard supernatant.
(15) Transfer precipitate to a tared glass-fiber filter with 15 ml water. Wash with successive portions of water, ethanol and ether.
(16) Dry, cool, weigh, mount and count.

Cesium analysis

(17) Filter contents of distillation flask from Step 9 through a Buchner funnel using Whatman No. 41 filter paper. Transfer filtrate to a clean beaker. Reserve residue for strontium analysis (Step 28).
(18) Add 1 M $NH_2OH.HCl$ dropwise until the supernatant turns straw colored.
(19) Slowly add 1 g ammonium phosphomolybdate and stir for 30 min, using a magnetic stirrer. Allow precipitate sc settle for at least 4 hr, and discard supernatant.
(20) Slurry precipitate into a centrifuge tube. Centrifuge and discard supernatant.
(21) Wash precipitate with 20 ml 1 N HCl, and discard wash solution.
(22) Dissolve precipitate by dropwise addition of 3–5 ml 6 N NaOH. Heat over a flame for several min to remove ammonium ions. (Moist pH paper turns green as long as NH_3 vapors are evolved.) Dilute to 20 ml with water.
(23) Add 10 ml 3 M $CaCl_2$ and adjust to pH with 6 N HCl tc precipitate $CaMoO_4$. Stir, centrifuge and filter supernatant through Whatman No. 41 (or equivalent) into a 50-ml centrifuge tube.
(24) Wash the precipitate remaining in the centrifuge tube with 10 ml water; filter through the same filter paper, and combine the wash with the filtrate. Discard filter paper.
(25) Add 2 ml 0.1 M H_2PtCl_6 and 5 ml ethanol. Cool and stir in ice bath for 10 min.
(26) Transfer to a tared glass-fiber filter with water. Wash with successive portions of water, 1 N HCl and ethanol.
(27) Dry, cool, weigh, mount and count.

Strontium analysis

(28) Transfer residue and filter paper mass from Step 17 into a clean beaker and leach by boiling with 200 ml 4 N HNO_3. Collect leachate by Buchner funnel filtration through Whatman No. 41 filter paper.
(29) Leach filter mass by boiling again with 200 ml 4 N HNO_3 collecting leachate by Buchner funnel filtration. Combine leachates in a beaker and discard filter residue.
(30) Add 18 N NaOH to contents of beaker to make basic and precipitate carbonates from hot solution with 1.5 M Na_2CO_3. Digest and let cool to room temperature.
(31) Centrifuge in a bottle and dry carbonate precipitate in a 110°C oven for 30–60 min. Add 30 ml fuming nitric acid to the precipitate in an ice bath to dissolve carbonates and convert them to alkaline earth nitrates. Add 5 drops of H_2O_2 and digest several min, then cool again in an ice bath. Centrifuge and discard acid supernatant.
(32) Slurry the precipitate with 25 ml 16 N HNO_3, add 5 drops H_2O_2 and digest several min. Cool in an ice bath, centrifuge, and discard acid supernatant.
(33) Repeat Step 32, then dissolve precipitate with 10 ml water and add 0.5 ml 0.1 M $FeCl_3$.
(34) Heat to near boiling in water bath and add 6 N NH_4OH dropwise until $Fe(OH)_3$ precipitates.
(35) Cool, centrifuge, and transfer supernatant to a clean centrifuge tube. Discard precipitate. Record time as beginning of yttrium ingrowth. (Complete Steps 36–43 without delay, to minimize ingrowth of ^{90}Y.)
(36) Add 10 mg of barium carrier, 3 drops methyl red indicator, and adjust pH to near 5 with a few drops 1 N HCl. (Color change is from yellow to red.)
(37) Add 5 ml ammonium acetate buffer solution and heat in water bath.
(38) Slowly add 1 ml 0.5 M Na_2CrO_4. Stir, heat, centrifuge. Transfer supernatant to a clean centrifuge tube. Discard precipitate.
(39) Add 2 ml 15 N NH_4OH to the supernatant, heat in water bath, and slowly add, with stirring, 5 ml 1.5 M Na_2CO_3. Digest for a few minutes, cool, centrifuge and discard supernatant.
(40) Dissolve precipitate with 5 ml 1 N HCl, add 10 ml water, repeat Step 39.
(41) Wash the strontium carbonate precipitate with 20 ml water, and discard wash solution.
(42) Slurry the precipitate with a minimum of water and transfer to a tared stainless steel planchet. Dry under infra-red lamps.
(43) Cool, weigh and count immediately. Store planchet for suitable time for yttrium-90 ingrowth.

Yttrium separation

(44) After the period for ^{90}Y ingrowth, slurry the precipitate on the planchet with 2 ml water, and transfer to a centrifuge tube with the aid of a rubber policeman. To make the transfer quantitative, wash the residue from the planchet with a small amount of 1 N HNO_3. Dissolve the precipitate in the tube with sufficient 1 N HNO_3, and dilute with water to 10 ml.

(45) Add 1.0 ml yttrium carrier and stir.

(46) Boil to expel dissolved carbon dioxide; cool to room temperature.

(47) Replace in water bath and make basic with 2–3 ml 15 N NH_4OH. Stir and digest until the yttrium hydroxide precipitation is complete.

(48) Cool, centrifuge and decant supernatant into a 100-ml beaker. Record time of separation. This is the end of yttrium-90 ingrowth and the beginning of yttrium-90 decay.

(49) Dissolve precipitate in 1 ml 1 N HNO_3 and dilute with water to 10 ml.

(50) Reprecipitate yttrium by dropwise addition of 15 N NH_4OH. Centrifuge. Combine supernatant with solution in the 100-ml beaker (Step 48).

(51) Repeat Steps 49 and 50. Save the combined supernatants in the beaker for strontium activity and gravimetric yield determination, Step 59.

(52) Dissolve the $Y(OH)_3$ precipitate from Step 50 with 2 ml 1 N HNO_3 and dilute to 5 ml with water.

(53) Slowly add 5 ml sat. $H_2C_2O_4$, with stirring, and digest in hot water bath for 10 min.

(54) Cool in an ice bath to room temperature.

(55) Centrifuge and discard supernatant.

(56) Wash precipitate twice with 10 ml hot water and discard wash solutions.

(57) Filter the yttrium oxalate on a tared glass-fiber filter. Wash with hot water and ethanol.

(58) Dry, cool, weigh, mount and count the ^{90}Y.

(59) Warm the combined supernatant solution from Step 51, add 5 ml 1.5 M Na_2CO_3 and digest for 10 min.

(60) Cool, centrifuge and discard supernatant.

(61) Wash the $SrCO_3$ with 15 ml water and discard wash solution.

(62) Slurry with a few ml water and transfer quantitatively to a tared stainless steel planchet. Dry under i.r. lamps.

(63) Cool, weigh and count immediately.